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TECHNICAL REPORT

Research in Soft X-Ray and Far Ultra-Violet Spectroscopy

E. Alexander and B.S. Fraenkel

Hebrew University

Jerusalem, Israel

Department of Physics

Annual Technical Report

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This technical report, on the research in the Ultrafar Vacuum UV and soft X-ray spectroscopy, covers the research done during the period of 1st January 1962 to 31st December 1962, in the X-ray and Ultrafar Vacuum UV Lab of this department, under Grant AF EOAR 62-33. The principal investigators are

E. Alexander

B.S. Fraenkel

On the subjects covered by the various paragraphs, the following physicists, and chemists are additionally working

§ 1. - A. Jacobs and U. Feldman

§ 2. - U. Feldman and A. Cohen

§ 3. - U. Feldman

§ 4. - A. Cohen

§ 5. - L. Cohen

The work described in § 6. was done by co-operation of B.S. Fraenkel with A. Weinreb, of the U.V. and Vacuum UV. Lab. of this department. With them are working

§ 6. - U. Leor and A. Teitelman.

A b s t r a c t

During the year 1962 the following research was carried out:

Building and adjustment of instruments for research in the Vacuum Ultra-Violet. Investigation into spectra obtained by the sliding spark method from the electrodes and from the insulator. High spectra of Cu in the 100-200 Å region, and the dependence of their intensities on electrical parameters. Determination of W(74) lines in the 100-200 Å region. Investigation into spectra of light elements in the 100-200 Å region. A method for obtaining absorption measurements of gases in the far ultra-violet has been developed.

## I. Instrumental

The work in the vacuum ultra-violet was done mainly with a grazing incidence spectrometer having a 2 meter curvature grating of about 30,000 lines per inch. Much effort was devoted to obtain the maximum resolution, as given by Mack, Stehn and Edlén<sup>(1)</sup>. Having obtained this

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(1) J.E. Mack, J.R. Stehn and Bengt Edlén, J.O.S.A. 22, 245 (1932)

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resolution, a full description of the instrument was written, which is being issued as a separate technical report.

The work in spectroscopy of multiply ionized atoms and of soft X-rays was done with a Pyrex glass spark chamber (or X-ray tube). During this year, however, two metal spark chambers (or X-ray tubes) were developed. The first is a replica of a usual Skinner<sup>(2)</sup> soft X-ray

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(2) H.W.B. Skinner, Phil.Trans.Roy.Soc.Lond. A239, 95(1940).

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tube worked out in brass, with liquid air traps made out of stainless steel. The vacuum obtained is not as good as the vacuum obtained with a glass tube, but good enough for work on spark spectroscopy in the far ultra violet. The vacuum obtained is of the order of  $10^{-6}$  mm. Hg. This tube was built because the glass tube broke several times, by overheating or strain.

The second tube was built in connection with soft X-ray spectroscopy. Investigation into the N bands of Gallium with standard equipment had to be interrupted because carbon contamination formed on the surface of the Gallium. A 1 m. grating-grazing-incidence-vacuum-spectrometer was built, with an ionization pump in place of the usual diffusion pump, to overcome this contamination. A new form of an X-ray tube, had to be built, however, for this new arrangement. This is because during bombardment of the anticathode with electrons, material evaporates away from it. Such material would be swallowed up by the usual diffusion pump, with no damage caused. In the case of an ionization pump, however, more caution is called for. Therefore a soft X-ray tube was built, with the purpose of trapping such material before it has a chance to be pumped away by the ionization pump. The usual soft X-ray tube is equipped with a liquid air trap near the anticathode. In this tube, however, the anticathode is surrounded by a cylinder-shaped liquid air trap. Two circular holes through this double walled container allow radiation to proceed to the slit system, and allow the anticathode to be adjusted to its proper place on the Rowland circle.

This new spectrometer has been tested and was found satisfactory regarding its vacuum. The first experiments on soft X-ray spectroscopy are being conducted.

## II. Spectra from the sliding spark

The method of the sliding spark<sup>(3)</sup> was tried in order to obtain

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(3) B. Vodar and N. Astoin, Nature, Lond. 166, 1029(1950)

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highly ionized spectra from metal electrodes. The three electrode method<sup>(4)</sup> using the sliding spark as a trigger was found out to fit

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(4) G. Balloffet, Ann.Phys., 5, 1256(1960)

J. Romand, G. Balloffet and B. Vodar, Spectrochim. Acta, 15, 454(1959)

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more to this purpose. However, before abandoning the two electrode sliding spark, some of its properties were investigated.

In the vacuum sliding spark method two electrodes are fastened on both sides of an insulator, and the spark between them develops at much lower voltage than the voltage needed for a spark between two electrodes in vacuum at the same distance without the insulator joining them. It was found out that the spectrum of O was more prominent than the other spectra obtained by this method. It was found out, further, that the best reproducibility was obtained with Alumina as an insulator. Other insulators investigated were Ge, Si, C, Quartz and China. Pieces of these insulators were chipped off during sparking, while the Alumina stood very well the high temperature developed during sparking. Therefore, our investigation on the nature of the spectrum obtained from the sliding spark was done mainly with Alumina as insulator.



The results of these investigations concern three different spectra:

1) The oxygen spectrum. 2) The aluminium spectra. 3) The spectra from the electrodes. The break-down voltage was changed with the number of sparks so that the energy which flowed through the electrodes in each exposure was kept constant. The range of voltages was between 6 K.V. to 15 K.V. The experiments were performed in the spectral range of 100 Å to 300 Å.

The O spectra O IV, O V, and O VI, were obtained in a more or less constant intensity. When Alumina pieces were used during many exposures, and were kept minimum time in air, the O lines weakened very considerably. This indicates that the O spectrum comes from the oxygen layers formed on the surface of the insulator and not from the bulk material.

The Aluminium spectrum obtained was mainly Al IV. The electrodes used were Cu. The Cu spectrum obtained was Cu VII, VIII and IX. Below a certain voltage the intensity of the Aluminium was much greater than the intensity of the Cu. Above this voltage, the intensity of the Cu lines was much greater than the intensity of the Al lines. This inversion voltage was, in the case of Cu electrodes on Alumina, in the geometrical arrangement used, about 10 K.V. The intensity of the Al lines was constant below 10 K.V. with the varying voltage. Also the intensity of the Cu lines was constant above 10 K.V., with the varying voltage.

The same experiment was repeated with Al electrodes. Below the inversion voltage a spectrum of Al IV was obtained. Above the inversion voltage, spectra of Al V, VI and VII, were obtained. The intensities behaved with varying voltage as described in the case of Cu electrodes.

### III. High spectra of Cu in the 100 - 200 Å region

It has been our intention to look into the spectrum of Cu IV. However, the grating employed in the spectrometer is a Siegbahn glass grating. Therefore, when used in the grazing incidence position, its reflecting power for wavelengths longer than 500 Å is very weak. A spectrum of Cu IV, however, should appear at a much longer wavelengths.

However, the higher spectra of Cu are obtained easily. The resonance lines of Cu VII at 200 Å are the only Cu lines known in this region. Three groups of lines, observed at lower wavelengths, are obtainable with the triggered spark method, at 5 to 18 K.V., with about 500 sparks and half microfarad. The lines are grouped according to their change in intensity with the changing voltage. Thus, two distinct groups of lines, whose intensities change relatively to one another and relatively to the Cu VII lines, begin at about 140 Å and 160 Å.

The isoelectronic spectra of the elements lighter than Cu VIII and Cu IX are known. It is therefore possible to calculate these spectra isoelectronically and it turns out that the spectra of Cu VIII and IX should begin at the wavelengths indicated.

About 150 lines of the group which belongs to the Cu VIII spectrum were registered. The isoelectronic calculation to determine the ground states of Cu VIII has been carried out. Classification of the lines is now in progress. Classification of the lines of Cu IX and of Zn IX and X will follow. This concerns the lines coming from transitions in the ground states of these spectra only.

#### IV. Tungsten lines in the 100 - 300 Å region

While looking for elements having a continuous background for absorption measurements, the spectrum of W(74) was obtained in the 100-300 Å region. An unclassified tungsten spectrum was published<sup>(5)</sup>. This spectrum was

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(5) R.I. Vasiliev and A.V. Yakovlieva, Optika i Spektroskopiya 5, 620(1958)

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obtained with  $1/3$  microfarad and 50 K.V. Our spectrum, obtained at 18 K.V. and with  $1/2$  M.f., showed more lines in this region (300 lines as against about 30 lines in the mentioned work). It was therefore thought worthwhile to measure these wavelengths with the help of standard lines in this wavelength region, which are the unclassified Cu lines<sup>(6)</sup>. This work is being carried out now.

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(6) P.G. Kruger and F.S. Cooper, Phys. Rev. 44, 826(1933)

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#### V. Spectra of light elements

A systematic research of lines in the 100-200 Å region of light elements is going on. At present carbon lines of the fifth and sixth spectrum are being looked for in this region. For this research a big, 8 microfarad capacitor, is used, so that the material is at a high temperature during sparking.

#### VI. Absorption Spectroscopy of gases in the far ultra-violet

Absorption spectroscopy of gases in the ultrafar U.V., under defined conditions, is a problematic task. As no windows may be used, the usual technique would be to fill the spectrometer with the gas to be investigated, at a low pressure, of e.g.  $10^{-2}$  mm Hg. During the absorption experiment the pumps would have to be shut off. Influence of the gaseous substance, e.g., gaseous benzene, on the reflection power of the grating, would have to be ignored. The different distances from the grating of each point on the Rowland circle would have to be taken into account in determining the absorption coefficients.

As the absorption coefficients in the far ultra-violet of various substances are the only absorption coefficients in the whole optical range unknown up to now, effort has been put to obtain a method which would make absorption spectroscopy easier in this region.

The problem may be defined as how to allow a column of fixed shape of gas to pass through a volume of high vacuum without windows. This problem was solved for a number of substances. A square tube of  $1 \text{ cm}^2$  crosssection was put horizontally through the spark chamber, so that it protruded both sides of the spark chamber. The direction of this tube is perpendicular to the slit system and perpendicular to the direction of the slit. It is set in-between the slit and the place of the spark, near the positive electrode. Two 1 mm wide slits, on both sides of the square tube, allow the light from the spark to pass through the tube to the slit system, and from there to the grating. Both ends of the square tube are connected to glass containers. In one container is the substance, the absorption of which is to be investigated, in the liquid or solid phase. For this purpose, the container is kept at a certain fixed temperature - at  $0^\circ\text{C}$  in the experiments to be described. The other container is kept at a very cold temperature, liquid air temperature in the experiments to be described.

As the spark chamber is kept in a high vacuum state, the second container acts as a special pump for the material of the first container. The mean free path of the molecules of the substance under investigation is, at about  $10^{-2} \text{ mm Hg}$ , much bigger than 1 mm, so that there is a good chance that only very little of this material escapes through the double slit in the square tube.

This experiment was tried out successfully with benzene and water, where practically no material was lost to the main vacuum during the experiment. Several hours of this differential pumping moved about 50 - 100 cc of the material from one side to the other. Absorption spectroscopy in the near ultra-violet gave reproducible absorption curves. In benzene, some measurements have been made upto now in the far ultra-violet, indicating that below  $200 \text{ \AA}$  the absorption is much smaller than in the  $200 - 300 \text{ \AA}$  region. Also, it became immediately clear that this method will be a powerful tool in separating high orders of short wavelength lines from long wavelength lines, i.e., it will solve partially the problems of filters in the vacuum ultra-violet.

However, it was found out that substances whose melting point is much lower than room temperature, e.g. alcohol, lost about 10 to 30 percent of the material involved to the main vacuum system. This problem is being attacked now from various angles.

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